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4. Title of the invention

EXTRACTION ROUTE FOR TiO<sub>2</sub> AND ALUMINA FROM  
BAUXITE AND BAUXITIC RESIDUES, AND TITANIFERROUS  
DEPOSITS AND WASTES

5. Name of your agent (if you have one)

URQUHART-DYKES & LORD LLP

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## Description

Claim(s) 2

Abstract 1

Drawing(s) 3

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### 1. Present scenario :

a. **Alumina extraction** : The existing process technologies used for extraction of alumina and  $TiO_2$  generates large amounts of hazardous wastes, which are disposed as landfills. The management of wastes and landfill sites is a major problem for these industries due to increasingly stringent environmental regulations. The Bayer process used for the extraction of alumina from bauxite ores is well documented and a large volume of literature is available on this process [1-5]. The process generates large amount of highly alkaline residue called red mud. The amount of residue generated strongly depends on the mineralogy, which determines its processing condition [6]. The presence of reactive silica (kaolinite) and other impurities also influence the process parameters and the amount of red mud generated. The disposal of large volume of highly alkaline red mud has become a major problem to alumina industries/refineries. The percolation of alkaline leachate into surface and ground water and dry dust from improperly maintained landfill sites create severe environmental problem. Disposal of red mud therefore creates a major environmental problem in those regions where the land is available at a premium, and the costs for maintaining a landfill site is high. Nations where the environmental regulations are quite strict, the total cost of red mud disposal could be as much as \$5 per tonne, which can be at least 5% of the sale price of alumina (\$80-120 per tonne). The best value of extraction efficiency of alumina from the most suitable bauxite deposits and concentrates varies between 55% and 60%, which means at least 45% of alumina remains locked in the Bayer process residue.

Various techniques were developed for the recovery of alkali, alumina,  $Fe/Fe_2O_3$ ,  $TiO_2$ ,  $SiO_2$  and other materials from the red mud, and for manufacturing of bricks and refractories [7-11]. However, most of these techniques are uneconomical and hence not used commercially. In most alumina industries, the common practice for red mud disposal is deep thickening to recover alumina and soda followed by wet-slurry or dry storage [12,13]. Worldwide where permissible, red mud waste is also disposed at sea or river basins, however this practice is gradually curtailed due to long-term effects on marine ecology [2]. Dry storage method has several advantages over wet slurry storage but it needs initial high investment. In the dry storage method, after deep thickening, the slurry is solar dried in specially designed ponds. When full, the sites are capped, the land is restored for other usage, and a follow-up monitoring program is put in place to ensure environmental compatibility. However, annually about 16% of the red mud (~6 - 7 MT) is not disposed at proper disposal sites [6].

In Hungary and former Soviet Union some work on the reduction of iron oxides via reduction using natural gas and via smelting was also carried out. However the iron separation still remained a major problem for the recovery of titanium dioxide [7, 14].

b. **Titania extraction** : Ilmenite is the major source for Ti metal and  $TiO_2$ . It accounts for 90% of the world's Ti deposit, the remaining 10% being naturally occurring  $TiO_2$  known as rutile.  $TiO_2$  itself is an important mineral used as a pigment material particularly in paints, coatings, paper, printing inks, synthetic fibres and

pharmaceuticals. It is also used in glass and glass ceramics manufacture, electro-ceramics and in welding fluxes. World reserves of ilmenite are quite extensive and found primarily in massive deposits, or as beach sand in Africa, India, USA, Canada, the FSU, Malaysia, Australia, Brazil and Scandinavia. At present, ilmenite ( $FeO \cdot TiO_2$ ) is transformed into synthetic rutile ( $TiO_2$ ) by either chemical or thermal methods [2]. Iron can be leached from the ilmenite using hydrochloric or sulphuric acid leaving behind 90-96%  $TiO_2$  known as synthetic rutile. The leachate is a waste from ilmenite processing and is disposed of either in pond, or into the sea, leading to a major loss of iron. In a carbothermic reduction process, ilmenite is reduced in an electric-arc furnace at around  $1650^{\circ}C$  to produce Fe metal in the form of pig iron, and  $TiO_2$ -containing slag. Some FeO is deliberately left in the slag in order to reduce the melting point. The  $TiO_2$  content varies between 72 and 98%. A high-temperature chlorination and oxidation step or leaching with sulphuric acid (for low-grade slag) is then required to produce high quality  $TiO_2$ . [5-17] High quality  $TiO_2$  is also produced from rutile. Chlorination of the rutile is used to produce  $TiCl_4$ , which is then oxidised to produce  $TiO_2$ . In view of the environmental concerns in relation to the use of such substances as hydrochloric and sulphuric acids and the disposal of copious amounts of acid waste produced by these processes, it is essential that a new, more environmentally acceptable approach for the extraction of  $TiO_2$  be developed [2].

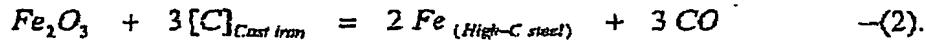
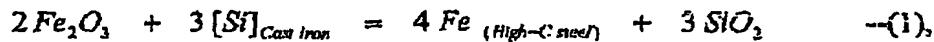
There has been a limited number of unsuccessful laboratory-scale investigation on the roasting of ilmenite with soda in a reducing atmosphere with carbon. However the yield of  $TiO_2$  was less than 50 percent. The major problem with this process was neither iron was separated in the metallic form, nor a leachable product was produced [2]. The reduction of ilmenite for making iron and  $TiO_2$  has also been studied extensively, however the extraction of  $TiO_2$  from reduced ore has been a major problem due to a fine distribution of Ti-oxycarbide and oxide phases [14].

## 2. NEW APPROACH

By considering the limitations of the Bayer and the  $TiO_2$  extraction processes and environmental issues related to disposal of red mud and acidic leachates, we developed a novel and economically viable zero-waste process for the extraction of alumina from bauxite, bauxite wastes, and other Al-rich raw materials, and  $TiO_2$  from ilmenite and Bayer waste. The materials flow diagrams are shown in Figures 1a and 1b and the process description are given below, which are very different approaches from conventional techniques. The main feature in the bauxite/bauxitic residue and the ilmenite processing is a *complete, efficient, and economical separation of large concentrations of iron oxides prior to alumina and  $TiO_2$  extraction*, which contribute to the waste and in the case of  $TiO_2$  contribute to the pigment quality.

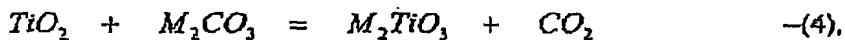
In the first stage, the iron oxide present in the ilmenite, bauxite ore and red mud is reduced to metallic iron. For bauxite and bauxitic waste processing, the ore and its waste, red mud along with lime and excess carbon is mixed and charged into the molten cast iron bath. For ilmenite, the ore is charged into a cast iron bath for promoting the reduction of iron oxides by carbon and silicon present in the molten cast iron. The excess carbon and

silicon in the cast iron reduce the iron oxides in the ore via reduction reaction, for example as shown in equations 1 and 2:



The reduction reaction, depending upon the mineralogy of ilmenite, red mud / bauxite smelted transforms cast iron into a high-carbon steel without increasing the need for additional energy consumption, since the reduction reactions between  $Fe_2O_3$  and Si and C, and the combustion of CO evolved are exothermic with a total  $\Delta H$  value of  $-305$  Kcal/mole. It is apparent from the Fe-C diagram<sup>[18]</sup> that by making 0.8 to 1.0% C steel from 4 to 4.5% C in cast iron, the melting temperature of high-C steel can be maintained at around  $1475^{\circ}C$  during the smelting process, at which the slag also remains molten. The carbon content in the steel can be further reduced, however this change in C-content needs an additional energy for maintaining the temperature of liquid metal bath. At the end of reduction step alkali compounds up to 50% by stoichiometric proportion are added in the slag. The reactions between the refractory oxides and alkali form alkali aluminate and alkali titanate as shown in equations 3 to 6.

For ilmenite processing, during the smelting stage, up to 50% by stoichiometric proportion alkali/alumina mixture or sodium aluminate is added in the cast iron bath to promote the formation of sodium aluminate based slag, which has a much lower melting point than either pure  $TiO_2$  or  $FeO\cdot TiO_2$  slag. The sodium aluminate slag is also a very good sink for any remaining unreduced iron oxides and silica formed. The presence of  $SiO_2$  however aids the separation of  $TiO_2$  at a later stage during water quenching. Excess silica can be fixed by the addition of an equivalent proportion of  $CaO$  in the sodium alumino-silicate slag. The addition of  $CaO$  changes the chemical association of alumina with silica to sodium oxide by forming sodium aluminate, whereas the  $TiO_2$  remains disproportioned in the pure form (i.e.  $TiO_2$  saturated slag) and in the calcium silicate phase.



where  $M = Na$  or  $K$ . The addition of lime may also be required to lock the silicate formed via reaction 1 and present in the red mud / bauxite ores. The lime addition reduces the excess consumption of alkali, which in its absence promotes the formation of alkali silicates. However, if an excess amount of lime is incorporated in the slag, it then aids a preferential partitioning of alumina between sodium-aluminate and calcium alumino-silicate. The formation of calcium alumino-silicate slag is not desirable for a complete extraction of alumina. The molten steel and slag is tapped separately. The excess alkali carbonate is added only before tapping of slag containing refractory oxides, which enhances the slag fluidity and facilitates a better separation of residual metal. Alkali dosing during slag tapping also minimizes the attack of alkali on refractory lining. Any unreacted

$\text{Al}_2\text{O}_3$  and  $\text{TiO}_2$  in the presence of excess alkali react to form complex aluminates and titanates, which are leached into an aqueous medium. The alkali aluminates are soluble in water, whereas the insoluble alkali titanates and silicates can be separated by filtration. The filtered residue is washed thoroughly to recover all the soluble aluminates and alkali compounds. Following filtration aluminium hydroxide is then precipitated from the filtered solution by bubbling  $\text{CO}_2$  gas generated during the reduction process. The hydroxide precipitate is washed thoroughly and calcined to produce alumina. The solid residue filtered after water leaching is digested in sulphuric acid and filtered to separate the undissolved silicates. By adjusting pH to 1 followed by the hydrolysis of filtered solution yields hydrated titanium oxide - a process, which is well known in the literature [2]. The filtered and washed precipitate is calcined to produce pigment grade  $\text{TiO}_2$ . The filtered solution is recycled in acid digestion step. The residue generated at the end of extraction process is mainly consist of silicates and have pH of 5 – 6, can be neutralized to pH = 7 and disposed as a soil conditioner containing  $\text{K}^+$  ions. The wastewater and filtered solution from various stages can be treated with  $\text{CO}_2$  gas, generated during the reduction and slag treatment steps, to recover alkali carbonate, which can be then recycled in the process.

The alternative method to high temperature reduction route has also been developed for extraction of  $\text{TiO}_2$  from ilmenite and other sources of titanium oxide. The process flow chart of new method is given in Figure 2. In this method, the ilmenite or  $\text{TiO}_2$  containing material is roasted with sodium / potassium carbonate together with 10 – 30 % alumina or other aluminium oxide containing materials in the temperature range from 800 to 1250°C. The amount of alkali carbonate was calculated based on the formation of alkali titanates and alkali aluminates. For better extraction efficiency up to 50 % excess alkali carbonates were used. After roasting, the roasted mass was leached in hot water and excess alumina and alkali were recovered by the treatment of  $\text{CO}_2$  generated in roasting step. The alkali carbonates and alumina were recycled in the first step. The filtered residue from water leaching was then leached with 5 – 20 % mineral acids ( $\text{HCl}$  /  $\text{H}_2\text{SO}_4$ ) solution to remove the iron compounds and decompose the sodium titanates into  $\text{TiO}_2$ . Adjusting the pH of the solution separated the dissolved iron. The filtered and washed residue consisted of 90 – 95 %  $\text{TiO}_2$ . Further purification of the  $\text{TiO}_2$  can be carried out either by low temperature roasting with alkali hydrogen sulphate or by electrolytic purification techniques. This new method substantially reduces the amount of wastes and the raw materials namely alkali carbonate and alumina can be recycled, which make this method economically viable. Utilizing it for regeneration of carbonates, which make process environmentally safe, also reduces the emission of green house gas,  $\text{CO}_2$ .

The following examples further illustrate the process of the present invention without any limitation of its scope.

#### **EXAMPLE I: Bauxite ore from Ghana**

The following inventive steps are involved in the extraction of alumina and other metal values.

1. The gray cast iron with 1 % Si and 4.2 % carbon was melting in the induction furnace. Bauxite ore from Ghana (approximate composition: 55% of  $Al_2O_3$ , 12 % of  $Fe_2O_3$ , 2% of  $TiO_2$ , 2 % of  $SiO_2$ , and moisture) was homogeneously mixed with lime and excess carbon and was added slowly in the molten bath. The temperature of the bath was adjusted to maintain metal and slag in molten condition.
2. The alkali carbonate (sodium or potassium) was added (20% excess to stoichiometric ratio) at the end of reduction operation and fluid slag was tapped.
3. The slag product was digested in hot water and filtered.
4. The filtrate was acidified with carbon dioxide to convert water-soluble sodium /potassium aluminate to  $Al(OH)_3$  precipitate.
5.  $Al(OH)_3$  was filtered out and calcined to produce pure  $Al_2O_3$ . The extraction efficiency of alumina was nearly 65%. Remaining alumina forms complex alumino-silicate products.
6. The filtrate was evaporated and sodium carbonate was recovered.
7. The residue containing titanium dioxide ( $TiO_2$ ) was mixed with 98%  $H_2SO_4$  for making a slurry. The ratio of  $H_2SO_4$  to residue was chosen in such a way that the weight ratio of  $H_2SO_4$  to  $TiO_2$  in the suspension produced by the hydrolysis was maintained between 2 to 2.5. Approximately 75 %  $TiO_2$  is extracted in the process.
8. The slurry obtained by digestion was dissolved in water.
9. Undissolved solid material containing silicates was removed completely by filtration.
10. Titanium oxide hydrate was precipitated from the filtered solution by hydrolysis in the temperature range 375 to 390K.
11. The titanium oxide hydrate was filtered off from the solution and calcined in the temperature range of 1050 to 1300K to produce pure titanium oxide.

#### EXAMPLE II (Red Mud)

Red mud containing (approximate composition: 46 %  $Fe_2O_3$ , 22 % of  $Al_2O_3$ , 8% of  $TiO_2$ , 8 % of  $SiO_2$ , 3-4 MgO and CaO and loss on ignition was 10-12 wt%) was charged with excess lime and carbon in the molten grey cast iron bath. The sodium / potassium carbonate (20% excess of stoichiometric ratio) was added in the slag before tapping. The experiments were carried out as described in the above examples by steps 2 to 12. The extraction efficiency for alumina was over 75 %.

The extraction efficiency of alumina from bauxite and red mud does not change when soda is replaced by potassium carbonate or potash. The red mud obtained in this case contains alkali in the form of potassium ions and does not have harmful effects of soda in red mud, which cannot be used as a fertilizer or soil conditioner. Below all the claims are referred in terms of alkali roasting of red mud and bauxite for the extraction of alumina, titania, and high carbon steel as by-product. Approximately 75 %  $TiO_2$  was extracted in the process. The residue consists of a complex alumino-silicates.

**EXAMPLE III ( Alkali roasting of ilmenite) :**

The following inventive steps are involved in extraction of  $TiO_2$  from ilmenite ore.

1. The ilmenite ore containing 63 %  $TiO_2$ , 32 %  $Fe_2O_3$ , 2 %  $Al_2O_3$ , was mixed with 10 % alumina and excess alkali carbonate and was roasted at  $1200^{\circ}C$  for 2 hours in air.
2. The alkali carbonate (sodium or potassium) was added (20% excess to stoichiometric ratio  $Na_2O : TiO_2$ ).
3. The roasted mass was leached in water and solution was filtered to separate residue.
4. The filtrate was acidified with carbon dioxide to convert water-soluble sodium /potassium aluminate to  $Al(OH)_3$  precipitate.
5.  $Al(OH)_3$  was filtered out and recycled in step 1.
6. The filtrate from step 5 was evaporated and sodium carbonate was recovered.
7. The residue containing titanium dioxide ( $TiO_2$ ) was leached with 5 % HCl solution.
8. The residue was filtered out and washed with acid solution and then with water.
9. Titanium oxide concentration in the residue was increased to more than 90 % at the end of this process.

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- 17) Patent : US 6,090,354, 2000.
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### Abstract

A novel zero-waste process is disclosed for extraction of alumina from different types of bauxite ores, red mud residues, and titanium dioxide from ilmenite deposits. In this process iron oxide is first reduced to metallic iron above the melting point of C-saturated cast iron alloy which yields a high-C iron alloy and an Al- and Ti- metal oxide rich slag, which is then treated with alkali carbonate to form alkali aluminates and titanates. The alkali aluminates are separated via water leaching from which the hydroxide of alumina is precipitated by bubbling CO<sub>2</sub>. The residue from water leaching is then treated with sulphuric acid and TiO<sub>2</sub> is precipitated via hydrolysis route. The process recovers most of the metal values and generates only small quantities of silicious residues with 4 – 5 pH, which can be used for soil conditioning.

**Claims**

We claim:

- 1) A process for the extraction of metal oxides, which comprises the steps of;
  - (i) Reduction of a mineral ore (illmenite/bauxite/clay) and alumina-containing residues in the molten cast iron bath followed by treatment with an alkali and/or alkali mixture;
  - (ii) Extracting the desired metal salt in the slag produced in step (i) using an aqueous and/or a dilute ammoniacal solution in aqueous media to separate water-soluble alkali aluminate from undigested metal oxide filter residue;
  - (iii) Precipitation of aluminium hydroxide from alkali aluminate solution by bubbling CO<sub>2</sub> gas or oxalic acid medium for maintaining a constant pH,
  - (iv) Calcining aluminium hydroxide formed in step (iii).
  - (v) Acidifying the metal oxide filter residue salt to produce a hydrated salt; and
  - (vi) converting the hydrated salt into an appropriate oxide.
- 2) A process according to Claim 1 characterised in that the mineral ore is bauxite.
- 3) A process according to Claim 1 characterised in that the mineral ore is red mud.
- 4) A process according to Claim 2 characterised in that the mineral ore is a mixture of bauxite and red mud.
- 5) A process according to Claim 1 characterised in that the mineral ore is illmenite.
- 6) A process according to Claim 1 characterised in that the carbonate is a carbonate of a Group Ia and Group IIa metals or a mixture thereof.
- 7) A process according to Claim 1 characterised in that the carbonate is an alkali metal carbonate or a mixture of alkali metal carbonates.
- 8) A process according to Claim 7 characterised in that the carbonate is selected from sodium carbonate and potassium carbonate.
- 9) A process according to Claim 8 characterised in that the carbonate is sodium carbonate.
- 10) A process according to Claim 8 characterised in that the carbonate is a mixture of sodium carbonate and potassium carbonate.
- 11) A process according to Claim 1 characterised in that the solvent used in the extraction process of step (ii) is water.

- 12) A process according to Claim 1 characterised in that the acid used in the acidification process of step (iii) is an inorganic acid.
- 13) A process according to Claim 12 characterised in that the inorganic acid is selected from hydrofluoric acid, hydrochloric acid, nitric acid, sulphuric acid and an acidic oxide or a mixture thereof.
- 14) A process according to Claim 13 characterised in that the acid is an acidic oxide.
- 15) A process according to Claim 14 characterised in that the acidic oxide is carbon dioxide.
- 16) A process according to Claim 1 characterised in that the process of step (iv) comprises calcining.
- 17) A process according to Claim 1 characterised in that the oxide produced is  $Al_2O_3$ .
- 18) A process according to Claim 1 characterised in that the oxide produced is  $Fe_2O_3$ .
- 19) A process according to Claim 1 characterised in that the oxide produced is  $TiO_2$ .
- 20) A metal oxide produced by a process according to Claim 1.
- 21) A metal oxide according to Claim 20 characterised in that the metal is selected from aluminium and titanium.
- 22) A metal oxide according to Claim 21 characterised in that the metal oxide is selected from  $Al_2O_3$  and  $TiO_2$ .
- 23) A metal oxide according to Claim 21 characterised in that the metal oxide is  $Al_2O_3$ .
- 24) A metal oxide according to Claim 22 characterised in that the metal oxide is  $TiO_2$ .
- 25) A process or a metal oxide substantially as described with reference to the accompanying examples.

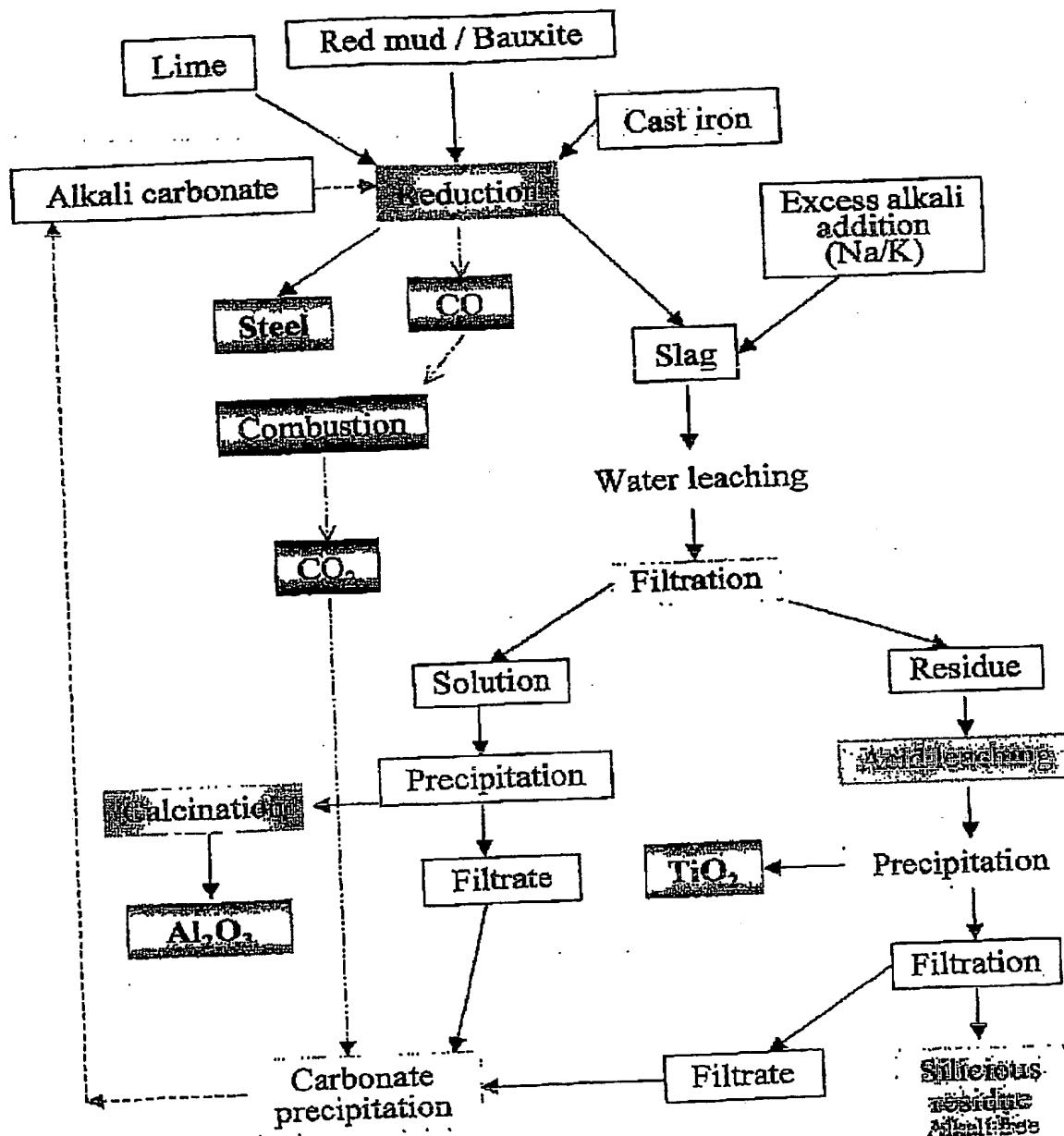


FIGURE 1 a : PROCESS FLOW CHART FOR PROCESSING OF BAUXITE / RED MUD.

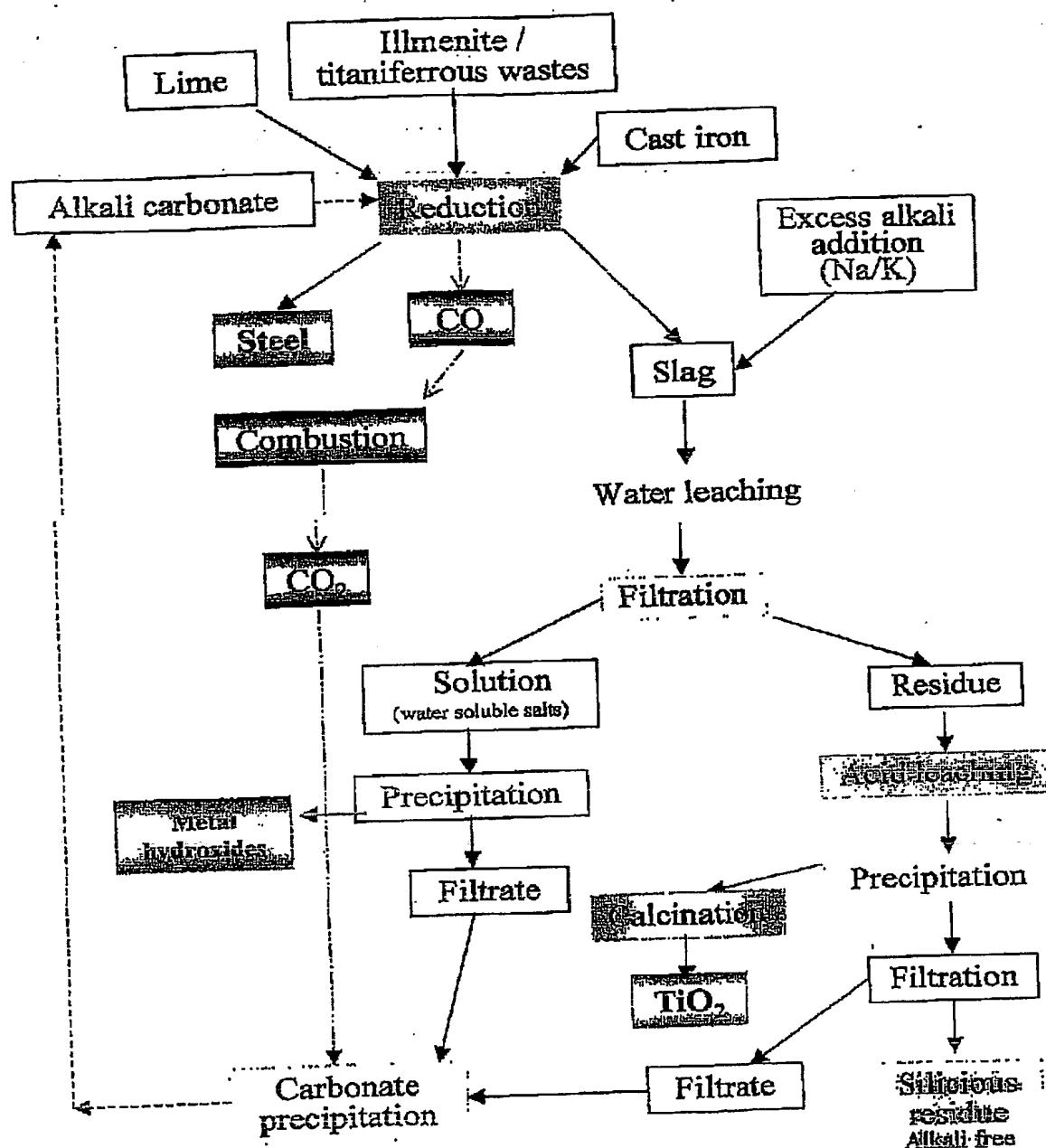


FIGURE 1 b : PROCESS FLOW CHART FOR PROCESSING OF ILLMENITE ORE.

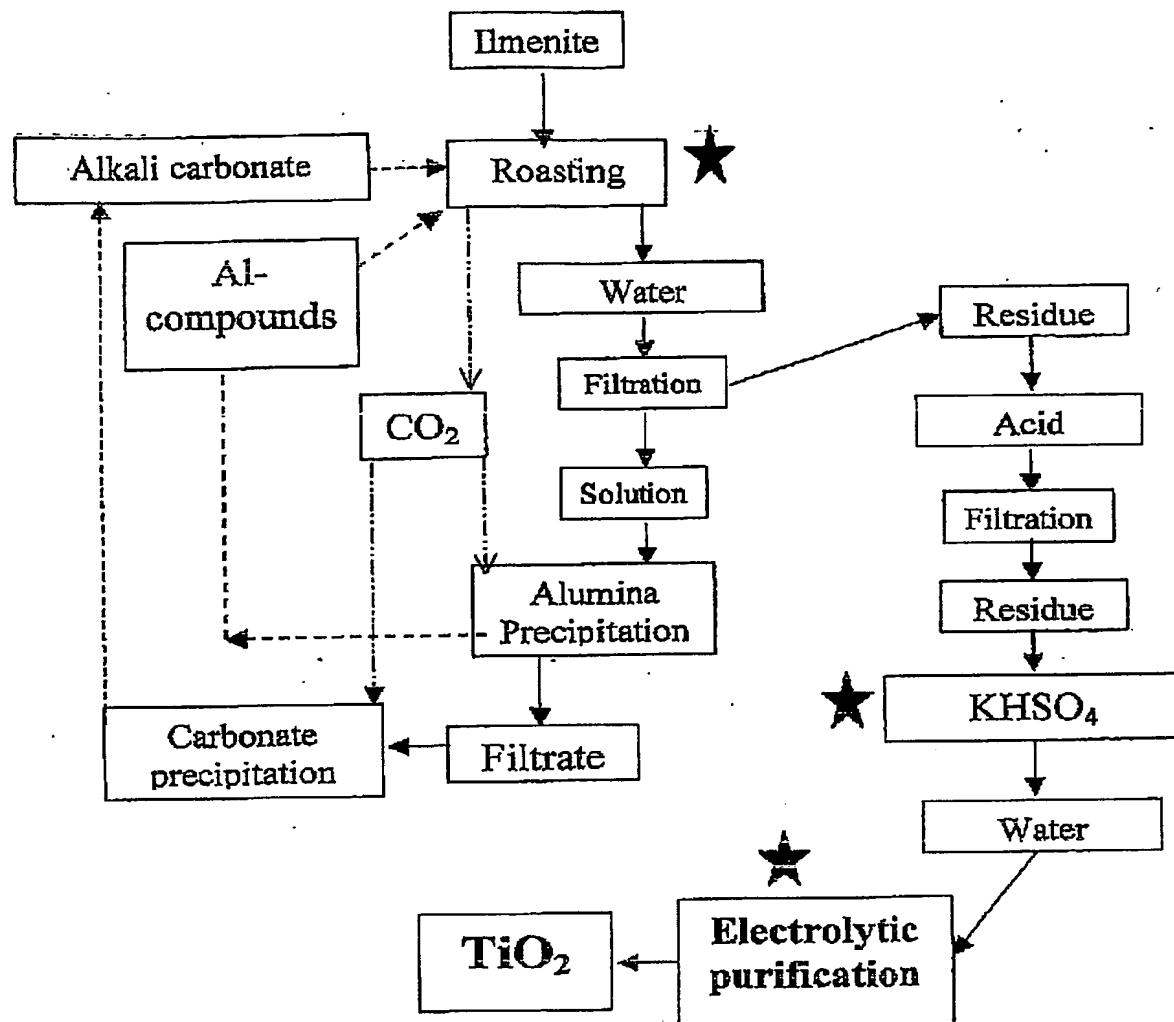


FIGURE 2 : PROCESS FLOW CHART FOR ALKALI ROASTING OF ILLMENITE ORE

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